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TECHNICAL REPORT 9122

EXPERIMENTAL METHOD FOR DETERMINATION OF THE RATE OF
EVAPORATION OF 2,4,6-TRINITROTOLUENE (TNT) AND 2,4-DINITROTOLUENE (2,4-DNT)

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U S ARMY BIOMEDICAL RESEARCH & DEVELOPMENT LABORATORY

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INTRODUCTION AND OBJECTIVES

According to our literature search the rate of evaporation of 2,4,6-trinitrotoluene (TNT) or 2,4-dinitrotoluene (DNT) has never been measured. Although the scientific literature does present measurements of the equilibrium vapor pressure of TNT^{1,2,3} these data have little use for describing the levels of TNT in air in actual scenarios.

In the real world, the TNT source (cm^2) is small and the reservoir into which it is released (cm^3) is large and is perturbed by ventilation. For these reasons, equilibrium vapor pressure has no application. If the rate of evaporation into air were known ($\text{g min}^{-1} \text{cm}^{-2}$) the actual concentration of TNT in air could be estimated. This problem was addressed by Griffy⁴ in modeling the TNT vapor for bomb detection. His description required the evaporation rate to estimate TNT concentration in air in a closed space.

Besides filling a gap in the literature that was long ignored, the empirical measurement of evaporation rate ($\text{g min}^{-1} \text{cm}^{-2}$) will be of use in estimating exposure levels to workers in the manufacture of TNT and DNT and contribute to validation of the model described by Griffy⁴ for detection of vapors from concealed explosives.

It was our objective to empirically measure the rate of evaporation of TNT and DNT into air under ambient conditions of air flow and temperature.

METHODS AND MATERIALS

CHEMICALS

Crude TNT was obtained from Volunteer Army Ammunition Plant, Tyner, Tennessee and purified by recrystallization from methanol, then hexane. The 2,4-DNT for this study was purchased from Aldrich Chemical Company, Inc. (Milwaukee, WI) and was purified by recrystallization from hexane. All solvents used for recrystallization and HPLC analysis were from Burdick and Jackson Laboratories (Division of Baxter Healthcare Corp., Columbia, MD).

HIGH PERFORMANCE LIQUID CHROMATOGRAPHIC (HPLC) ANALYSIS

The HPLC method employed was a simplification of a procedure published by Brueggemann⁵. The method was simplified by using an isocratic mobile phase because the samples contained few if any background interferences. The isocratic mobile phase provided adequate resolution of all the compounds of interest.

A liquid chromatographic system (Waters Chromatography Division, Millipore, Milford, MA) was used throughout the study. The system consisted of the following components: a model 6000A solvent delivery system, a model 721 programmable system controller, a model 730 data module, and a model 710B WISP autosampler. The UV detector was a spectroflow 783 programmable absorbance detector (Kratos Analytical Instruments, Ramsey, NJ).

Separation of the explosives was achieved with a ZorbaxTM ODS column (25cm x 4.6mm i.d., DuPont Instruments Co., Wilmington, DE). An isocratic mobile phase (60 percent methanol/water) was employed at a flow rate of 1.0 mL/min. The column effluent was monitored at 254 nm, 0.002 absorbance units full scale. The injection volume was 25 microliters.

METHOD FOR MEASUREMENT OF RATE OF EVAPORATION

The experimental method to measure the rate of evaporation consists of passing air at constant velocity across a measured surface of explosive at constant temperature. The explosive from the air is trapped in a solvent trap and its mass is measured.

The surface consisted of the inner wall of a pipe of cast explosive that is shown in Figure 1. The hollow cylinder of explosive is made by pouring molten TNT or 2,4-DNT into the annulus formed by a glass tube and a smaller, straight TeflonTM (PTFE) tube inserted along the center of the glass tube. The explosives were individually melted in a steam bath and individually poured into separate molds. When the explosives had cooled and solidified, the TeflonTM tube was gently withdrawn to create a hole of known diameter inside of a cylinder of solid 2,4-DNT or TNT. The case material is supported by the outer glass tube. Into each end of this pipe is inserted a short piece of the same PTFE tubing that was used to create the hole in the cylinder. These tubes form a tight connection and allow nitrogen to pass from a source of compressed nitrogen, through the pipe and then through a cold trap. To maintain a snug fit of the inlet and outlet tubes, each tube is inserted 1 cm into the pipe and this point of connection wrapped tightly with ParaFilmTM in order to prevent any leakage. As nitrogen moves between the inlet and outlet, it encounters a smooth surface of 2,4-DNT or TNT of known area. The effluent from the cast pipe passes through a glass trap fabricated from a 3-mL pipette shown in Figure 2. The trap contained 0.5 mL of methanol and was chilled in an ice bath. Preliminary experiments showed that a dry cold trap was not as effective as a cold trap containing methanol. To ensure that all of the effluent vapor was trapped, some experiments were carried out with two traps connected in series. From these experiments it was determined that a single trap was sufficient for collecting the vapors at most flow rates. The flow rate (mL/min) of nitrogen through the cast pipe and into the trap was monitored periodically during each run by a GilibratorTM model electronic bubble flowmeter (Gilian Instrument Corp., West Caldwell, NJ) connected to the outlet of the trap. After a measured time, the trap was disconnected and the methanol in the trap removed. The trap was rinsed twice with more methanol and the solution and rinses combined in a single vial. The amount of methanol was determined gravimetrically in order to obtain a better estimate of the sample volume. The concentration of TNT or 2,4-DNT in the methanol was determined by HPLC analysis. From the mass of TNT or 2,4-DNT found, mean flow rate of nitrogen through the apparatus and the length of time for a run, the rate of evaporation as grams per minute per square centimeter ($\text{g min}^{-1} \text{cm}^{-2}$) could be calculated.

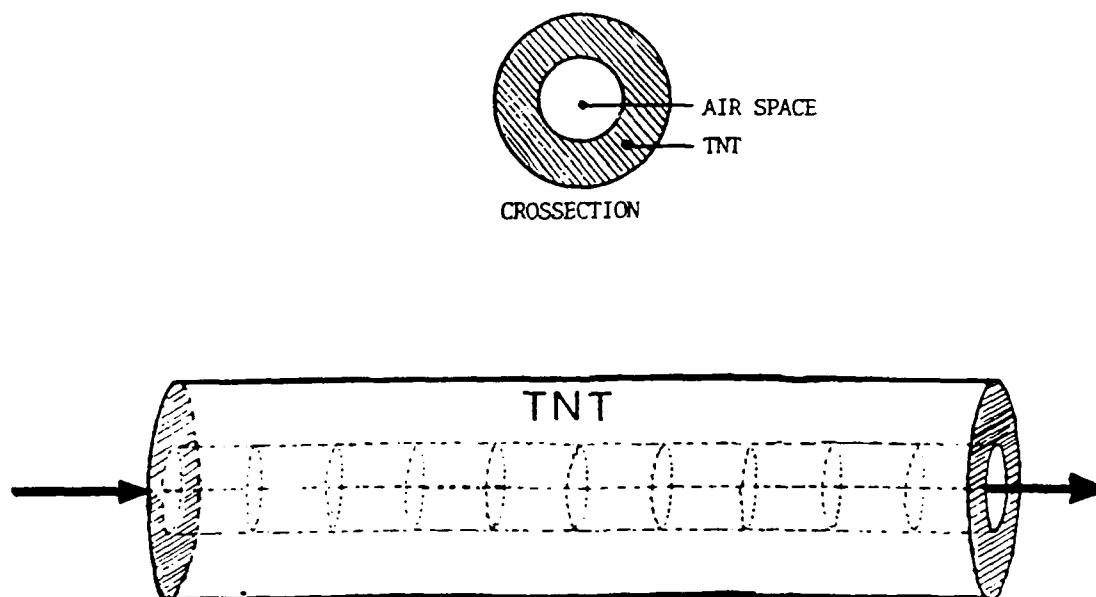


Figure 1. Cross section and side view of cylinder cast from molten TNT or 2,4-DNT. The inner surface serves as a measured area for evaporation and has an internal diameter of 0.4 cm.

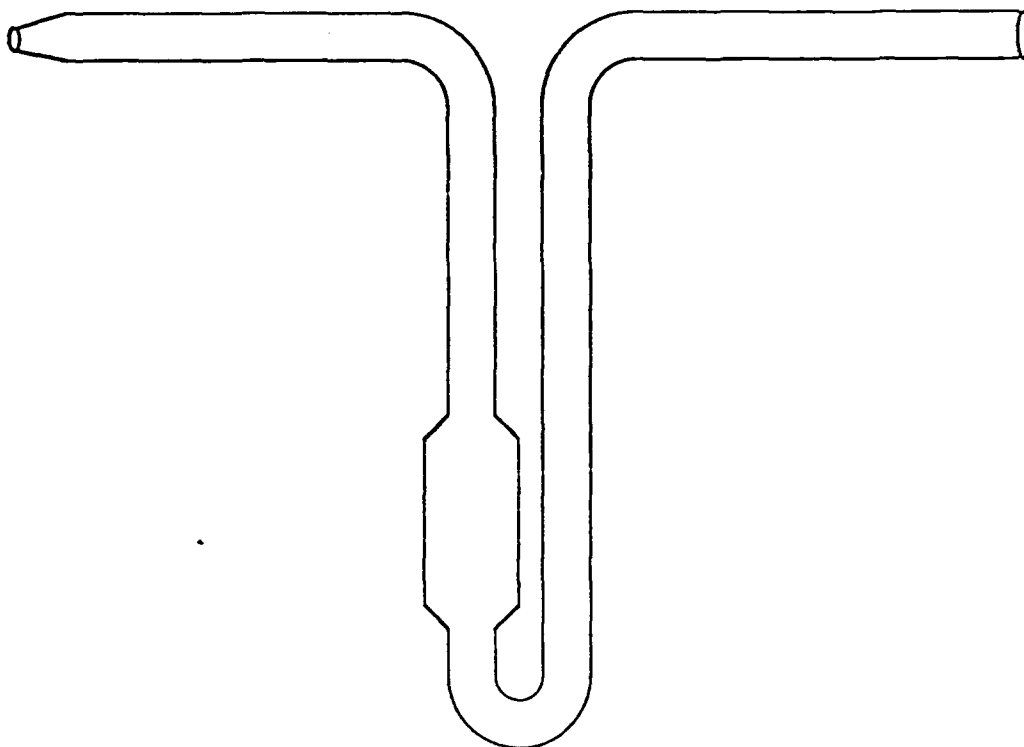


Figure 2. Drawing of the trap fabricated from a 3-mL pipette. This held 0.5 mL of methanol and was chilled in an ice bath. The effluent from the evaporating source entered through the blunt end via a 2-cm piece of PTFETM tubing and bubbled through the cold methanol while depositing the TNT or 2,4-DNT. The tip of the pipette-trap was connected via a TygonTM tube to a GilibratorTM model flow meter to determine the flow rate of nitrogen through the source.

RESULTS

Table 1 presents the actual data collected from a pipe of cast TNT from three different days. The surface area of the TNT in this case was 3.58 cm². Table 2 presents data from a cast pipe of 2,4-DNT. The surface area of 2,4-DNT was 3.41 cm². Figures 3 and 4 present the rate of evaporation of TNT and 2,4-DNT, respectively, as a function of velocity of nitrogen across the surface of the material. As would be expected, the rate of evaporation increases as the velocity of nitrogen increases until a point is reached at which the increase in the rate of evaporation levels off. This can be seen in Figure 4 for 2,4-DNT. The point of leveling off represents the maximum achievable evaporation rate. In the real world situation, where there would be little air movement across the surface of a concealed or stored explosive this maximum rate would only be obtained under extraordinary conditions, such as high winds due to extreme weather (i.e., hurricane, tornado). The more realistic rate of evaporation would be represented by the portion of the curve at the slower velocities.

The empirical data shown in Figure 3 for TNT was used by Griffy⁶ to validate his theoretical model, which in part estimates the rate of evaporation of TNT as a function of air velocity. Shown in Figure 5 is Griffy's theoretical curve which was derived from his model superimposed upon the empirical data from Figure 3. As can be seen the empirical data adheres closely to Griffy's theoretical model.

Reynolds numbers were calculated for both TNT and 2,4-DNT data sets and displayed in Tables 1 and 2. Reynolds numbers were calculated to determine if the flow through the cast pipes of TNT and 2,4-DNT was laminar or turbulent in nature. The calculated Reynolds numbers show that the flow through the cast pipes in both cases was well within the laminar flow region. We did not attempt to approach turbulent flow due to the limitations of our fabricated effluent trap. The high flow rates required to reach turbulent flow would have blown the methanol from our cold trap and thus reduced its trapping efficiency. If the flow were turbulent, it would have resulted in erratic and elevated evaporation rates. The data showed that at higher velocities the increases in evaporation rate was due to true evaporation and not to erosion from laminar flow across the surface of the cast pipes. The evaporation rates for 2,4-DNT and TNT were plotted versus their calculated Reynolds numbers (Tables 3 and 4) and are shown in Figures 6 and 7. These plots are very similar to the plots of evaporation rates versus velocity of nitrogen across the cast pipe surface. This is as expected because Reynolds number are directly related to the velocity of the gas across the surface.

TABLE 1. EXPERIMENTAL DATA FOR EVAPORATION OF TNT^a.

CODE	Run Time (min)	Air Flow (L/min)	Air volume collected (L)	Volume of methanol in (mL)	TNT in Methanol (mg/L)	Total TNT trapped (grams)	Conc. TNT in effluent air (g/L)	Evaporation rate TNT ₂ (g/min cm ²)	Velocity of air over TNT (cm/sec)	Comments
DAY 1										
B	6.66	0.467	3.11	1.45	0.036	5.20E-08	1.68E-08	2.18E-09	69	20-22 deg. C
C	6.00	0.438	2.63	2.15	0.05	1.07E-07	4.07E-08	4.98E-09	65	
D	5.00	0.438	2.19	1.90	0.025	4.75E-08	2.17E-08	2.65E-09	65	near detection limit
E	8.33	0.44	3.67	1.50	0.045	6.75E-08	1.84E-08	2.26E-09	65	
F	7.67	0.538	4.12	1.65	0.044	7.26E-08	1.76E-08	2.64E-09	79	
G	7.92	0.532	4.21	2.25	0.034	7.65E-08	1.81E-08	2.70E-09	78	
H	6.67	0.8	5.33	1.90	0.053	1.01E-07	1.88E-08	4.23E-09	118	
I	5.07	0.805	4.08	1.90	0.034	6.46E-08	1.58E-08	3.56E-09	119	
J	6.75	0.34	2.30	1.75	0.039	6.82E-08	2.97E-08	2.82E-09	50	
K	6.67	0.342	2.28	1.85	0.028	5.18E-08	2.27E-08	2.17E-09	51	near detection limit
L	8.80	0.18	1.58	1.95	0.028	5.46E-08	3.45E-08	1.73E-09	27	
M	5.83	0.175	0.94	2.00	0.025	5.00E-08	5.30E-08	2.39E-09	26	near detection limit
DAY 2										
A	25.36	0.087	2.21	1.91	0.044	8.40E-08	3.80E-08	9.25E-10	13	21 deg. C
B	23.67	0.092	2.17	2.01	0.065	1.31E-07	6.03E-08	1.55E-09	14	
C	14.33	0.216	3.14	1.91	0.051	9.74E-08	3.10E-08	1.90E-09	32	
D	11.17	0.272	3.04	1.92	0.049	9.41E-08	3.09E-08	2.35E-09	40	
E	11.38	0.308	3.51	1.88	0.056	1.05E-07	3.00E-08	2.58E-09	45	
F	13.17	0.395	5.21	1.72	0.05	8.58E-08	1.65E-08	1.82E-09	58	
G	8.67	0.474	4.11	1.86	0.039	7.25E-08	1.76E-08	2.33E-09	70	
H	10.83	0.595	6.45	1.89	0.046	8.69E-08	1.35E-08	2.24E-09	88	
I	8.47	0.605	5.12	1.98	0.053	1.05E-07	2.05E-08	3.46E-09	89	
J	7.67	0.617	4.73	2.09	0.038	7.93E-08	1.68E-08	2.89E-09	91	
K	6.78	0.753	5.11	1.74	0.08	1.40E-07	2.73E-08	5.76E-09	111	
L	15.57	0.139	2.17	1.75	0.05	8.75E-08	4.04E-08	1.57E-09	21	
M	12.2	0.387	4.72	1.87	0.051	9.54E-08	2.02E-08	2.18E-09	57	
DAY 3										
A	21.66	0.105	2.29	1.72	0.0264	4.54E-08	1.98E-08	5.85E-10	16	20.5 deg. C
B	18.7	0.121	2.26	1.76	0.0264	4.65E-08	2.06E-08	6.94E-10	18	
C	16.17	0.13	2.1	1.9	0.0266	5.05E-08	2.4E-08	8.72E-10	19	
D	15.4	0.141	2.17	1.88	0.0249	4.68E-08	2.15E-08	8.49E-10	21	
E	15.63	0.16	2.5	1.76	0.0364	6.41E-08	2.56E-08	1.15E-09	24	
F	15.57	0.196	3.05	1.79	0.0329	5.89E-08	1.93E-08	1.06E-09	29	
G	14.17	0.414	5.86	1.66	0.0475	7.88E-08	1.34E-08	1.55E-09	61	
H	9.67	0.516	4.99	1.52	0.0349	5.30E-08	1.06E-08	1.53E-09	76	
I	10.4	0.598	6.22	1.57	0.0401	6.29E-08	1.01E-08	1.69E-09	88	

^a TNT SOURCE IS A CAST PIPE WITH 3.58 CM² AREA AND A 0.113 CM² CROSSSECTION.

TABLE 2. EXPERIMENTAL DATA OF EVAPORATION OF 2,4-DNT^a.

CODE	Run Time (min)	Air Flow (L/min)	Air volume collected (L)	Volume of methanol (mL)	Conc. 2,4-DNT in Methanol (mg/L)	Total 2,4- DNT trapped (g)	Conc. DNT in effluent air (g/L)	Evaporation rate 2,4-DNT (g/min cm ²)	Velocity of air (cm/sec)
A	6.77	0.237	1.60	2.00	0.46	9.20E-07	5.73E-07	3.98E-08	35
B	11.58	0.238	2.76	1.80	0.92	1.65E-06	5.98E-07	4.18E-08	35
C1b	20.00	0.233	4.40	1.81	1.78	3.23E-06	7.34E-07		34
C2b	20.00	0.233	4.40	1.47	0.04	5.86E-07		5.59E-08	34
D	11.68	0.257	3.00	1.74	1.13	1.97E-06	6.57E-07	4.95E-08	38
E	5.13	0.253	1.30	1.97	0.48	9.41E-07	7.24E-07	5.38E-08	37
F	4.00	0.748	3.00	1.33	0.912	1.21E-06	4.03E-07	8.87E-08	110
G1b	6.20	0.686	4.25	2.13	0.813	1.73E-06	4.07E-07		101
G2b	6.20	0.686	4.25	2.04	0.04	7.95E-08		8.56E-08	101
H	6.63	0.738	4.89	2.03	0.943	1.91E-06	3.91E-07	8.45E-08	109
I	4.25	0.945	5.20	2.02	0.649	1.31E-06	2.52E-07	9.04E-08	139
J1b	5.27	0.907	4.77	1.93	0.816	1.57E-06	3.69E-07		134
J2b	5.27	0.907	4.77	1.92	0.101	1.93E-07		9.81E-08	134
K	42.15	0.089	3.75	1.69	2.743	4.63E-06	1.25E-06	3.22E-08	13
L	115.00	0.086	9.89	1.91	4.743	9.07E-06	9.16E-07	2.31E-08	13
M	8.33	0.42	3.50	2.00	1.19	2.35E-06	6.72E-07	8.27E-08	62
N	10.12	0.424	4.29	2.25	1.24	2.79E-06	6.50E-07	8.08E-08	63
O	10.32	0.534	5.51	1.65	1.822	3.01E-06	5.46E-07	8.55E-08	79
P	8.00	0.578	4.62	1.67	1.516	2.53E-06	5.42E-07	9.26E-08	85
QC	20.00	0.57	11.02	1.66	0.378	6.26E-07	5.66E-08	ND	-
Rd	BLANK			2.00	ND	0.00E+00	0.00E+00	ND	-

^a 2,4-DNT SOURCE IS A CAST PIPE WITH 3.41 CM² SURFACE AREA AND A 0.113 CM² CROSSSECTION

^b 2 TRAPS IN SERIES

^c EMPTY TRAP

^d SOLVENT BLANK

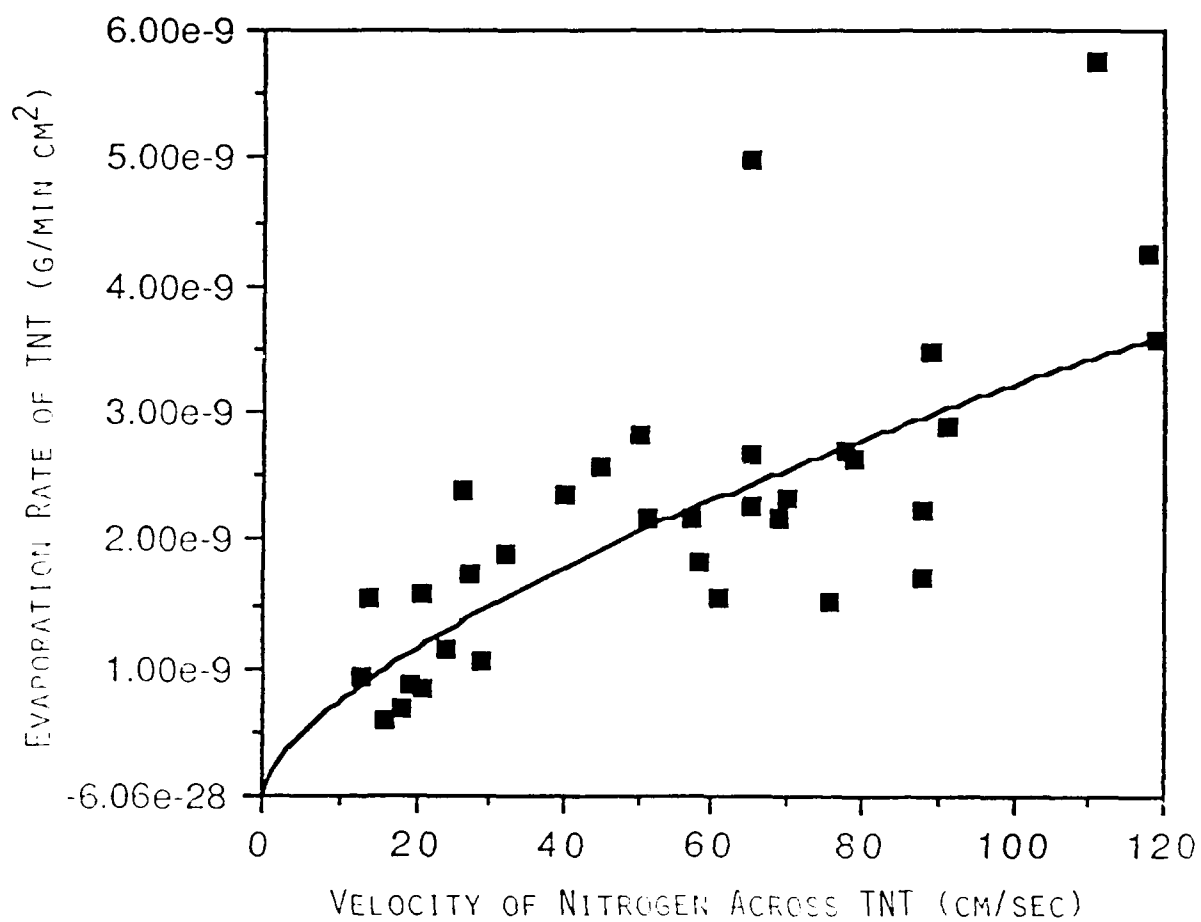


Figure 3. Plot of rate of evaporation of TNT at 23-25°C versus the velocity of nitrogen across the surface of TNT.

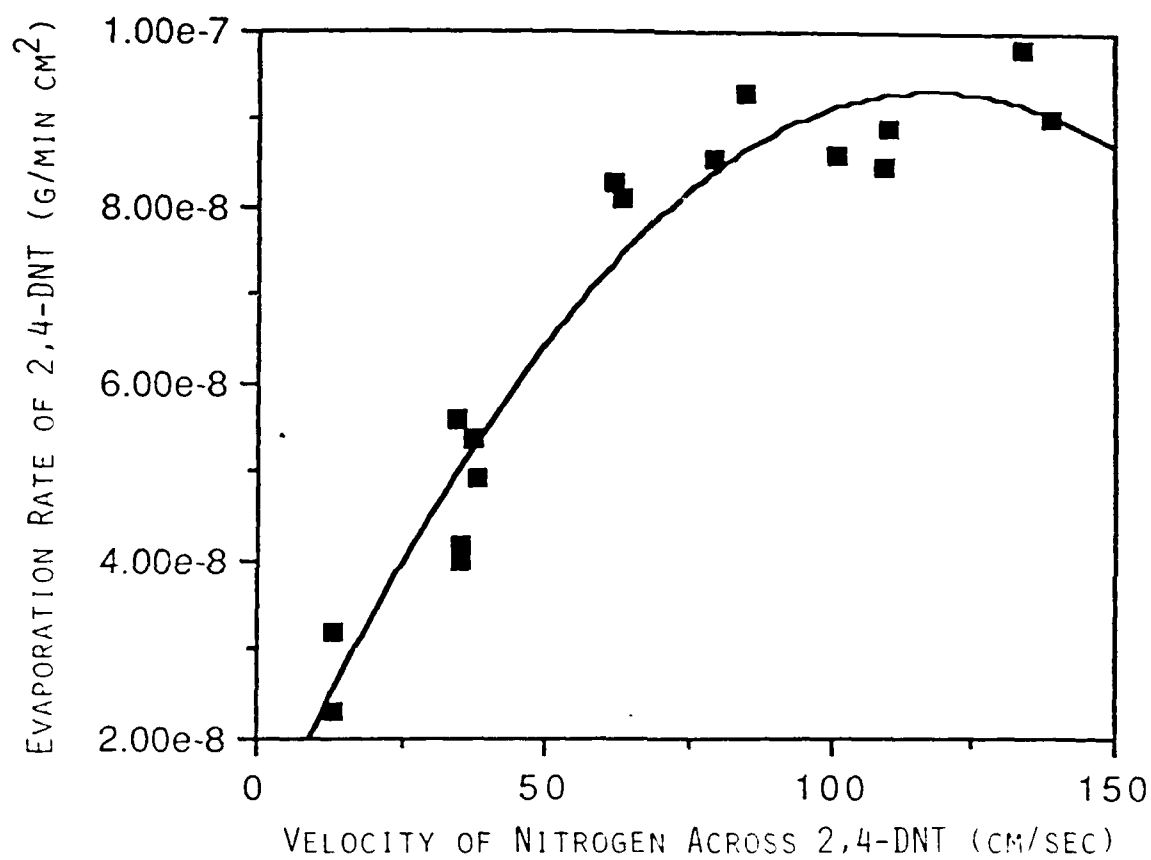


Figure 4. Plot of rate of evaporation of 2,4-DNT at 23-25°C versus the velocity of nitrogen across the surface of 2,4-DNT.

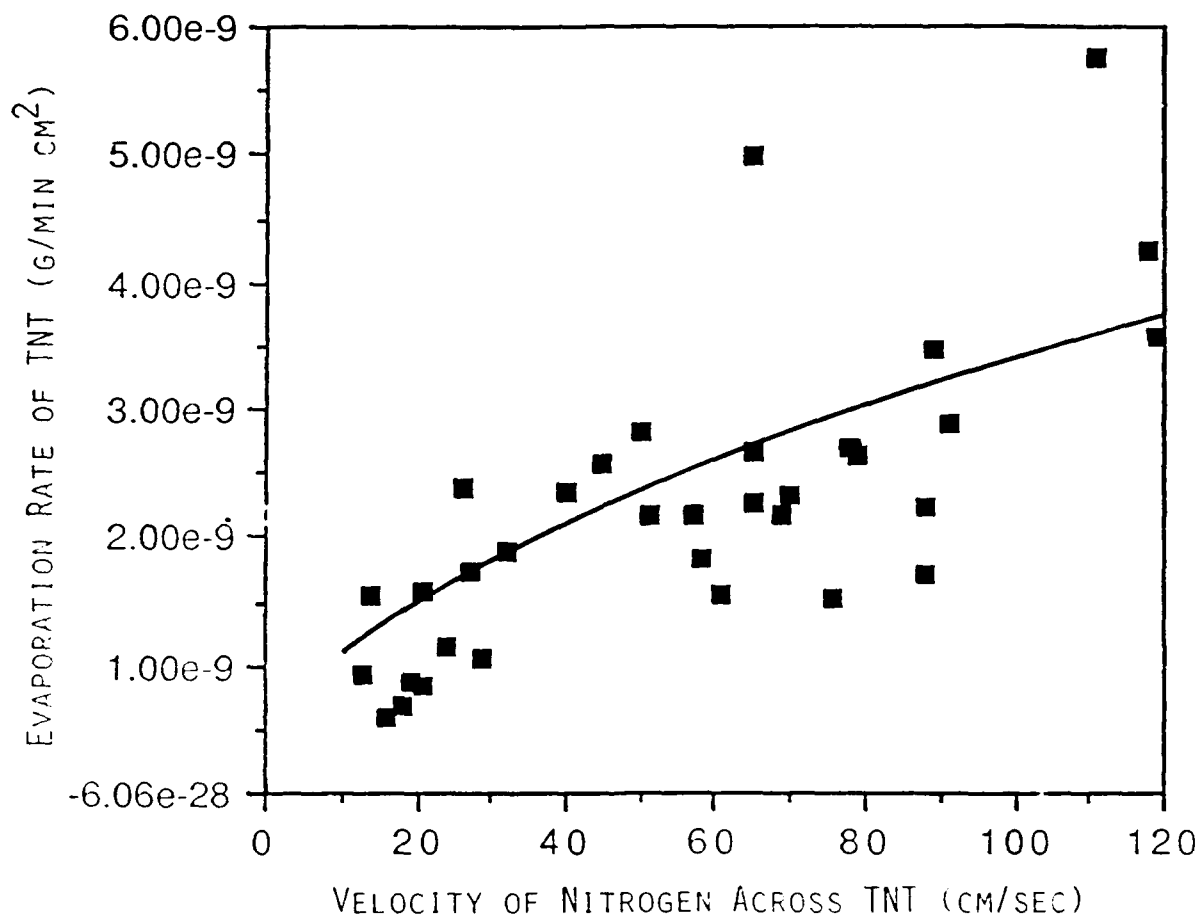


Figure 5. Griffy's theoretically derived curve superimposed upon the empirical data for TNT from figure 3.

TABLE 3. REYNOLDS NUMBERS FOR TNT DATA.^a

<u>CODE</u>	<u>REYNOLDS NUMBER</u>
DAY 1	
B	176
C	166
D	166
E	166
F	202
G	199
H	301
I	304
J	128
K	130
L	69
M	66
DAY 2	
A	33
B	36
C	82
D	102
E	115
F	148
G	179
H	225
I	227
J	232
K	284
L	54
M	146
DAY 3	
A	41
B	46
C	49
D	54
E	61
F	74
G	156
H	194
I	225

^a TNT SOURCE IS A CAST PIPE WITH 3.58 CM² SURFACE AREA, A DIAMETER OF 0.4 CM AND A 0.113 CM² CROSSECTION.

TABLE 4. REYNOLDS NUMBERS FOR 2,4-DNT DATA.^a

<u>CODE</u>	<u>REYNOLDS NUMBER</u>
A	94
B	94
C1 ^b	91
C2 ^b	91
D	102
E	100
F	296
G1 ^b	272
G2 ^b	272
H	293
I	374
J1 ^b	360
J2 ^b	360
K	35
L	35
M	167
N	169
O	212
P	228
Q ^c	ND
R ^d	ND

^a 2,4-DNT SOURCE IS A CAST PIPE WITH 3.41 CM² SURFACE AREA, A DIAMETER OF 0.4 CM AND A 0.113 CM² CROSSECTION

^b 2 TRAPS IN SERIES

^c EMPTY TRAP

^d SOLVENT BLANK

ND NOT DETERMINED

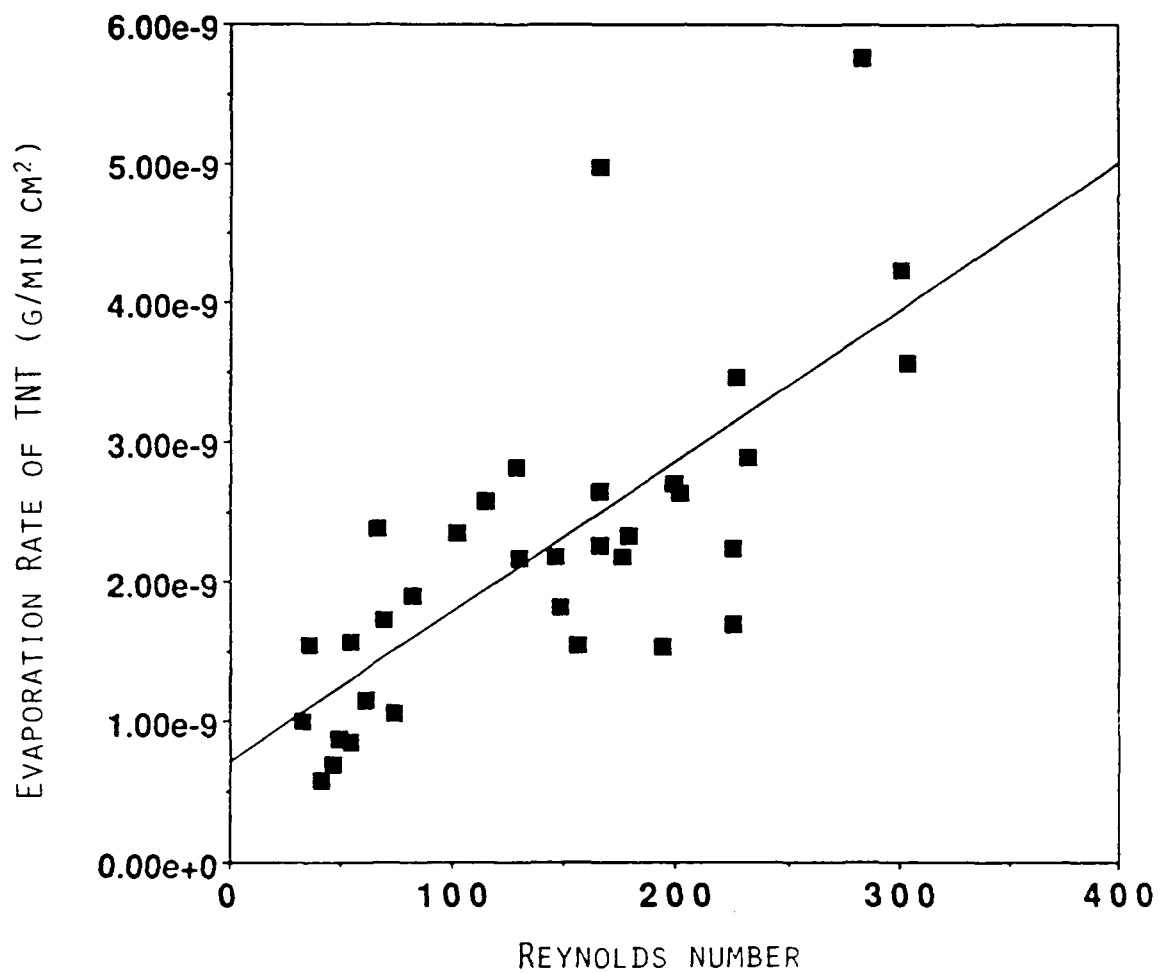


Figure 6. Plot of rate of evaporation of TNT at 23-25°C versus the calculated Reynolds numbers for nitrogen flowing across the surface.

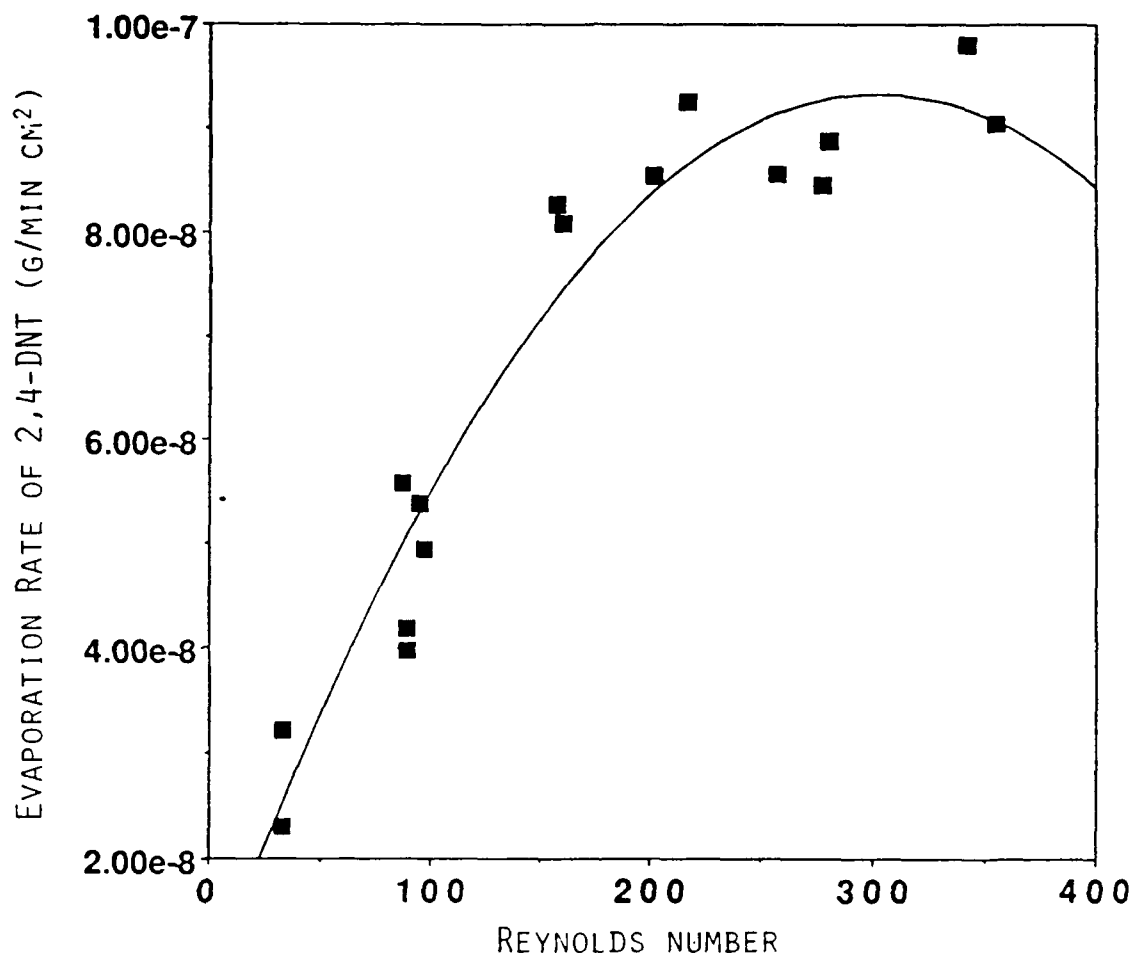


Figure 7. Plot of rate of evaporation of 2,4-DNT at 23-25°C versus the calculated Reynolds numbers for nitrogen flowing across the surface.

CONCLUSION

For the first time, the rate of TNT and 2,4-DNT evaporation into air ($\text{g min}^{-1} \text{cm}^{-2}$) under ambient conditions has been measured. This measurement allows for the validation of the Griffy model for estimating TNT in air for detection of explosives.

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